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FINAL REPORT - FY 1999 ARO DURIP PROJECT "Nanoworkbench for Analysis, Manipulation, and Excitation of Individual Nanostructures."

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1. Summary

The nanoworkbench project has been partially funded by ARO/DURIP in March 1999. The construction of this novel system includes a unique multiple tip STM device, coupled with an SEM and with an Auger analyzer for the study of single nanostructures in ultrahigh vacuum. It is split into several development stages. The first stage is supported by shared contributions of the University of Pittsburgh and ARO/DURIP.

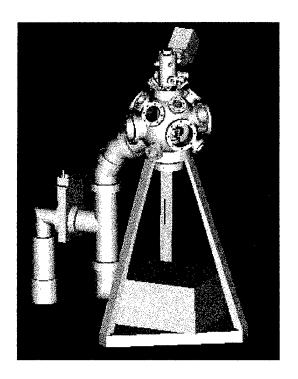
Two students and Dr. Joachim Ahner are working full time on this project. The construction in the first stage (March 1999 – March 2000) has been focused in the following directions:

- Designing and building of a vibration isolated ultrahigh vacuum system.
- Software development for the STM/SEM electronics.
- Exploration of novel tip preparation procedures. Narrow tip shapes are essential for the performance of the multiple tip STM.

We have obtained first experimental results in the STM tip production and SEM imaging of correlated double tip STM motion.

2. The UHV System

Figure 1a and Figure 1b show pictures of the ultrahigh vacuum apparatus designed and built to house the nanoworkbench. It is mounted on vibration isolators and is pumped by a high speed ion pump and a turbopump. A FEI SEM column is mounted on the top, and the channeltron detector of secondary electrons is mounted on the left-hand side of the spherical chamber. A support platform for the sample and for the multiple STM probes is mounted inside the chamber, and may be inserted from the lower flange. For analysis of residue and processing gases, we modified and computerized an existing UTI quadrupole mass-spectrometer (QMS). Figure 2 shows the developed Labview program for the QMS control.



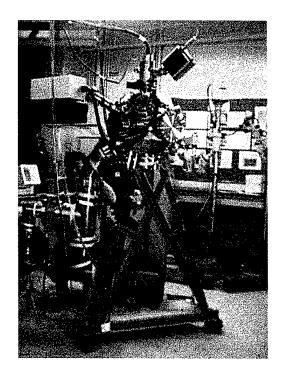


Figure 1. UHV nanoworkbench chamber showing SEM and STM platform entry port. (a) Design and (b) actual working system.

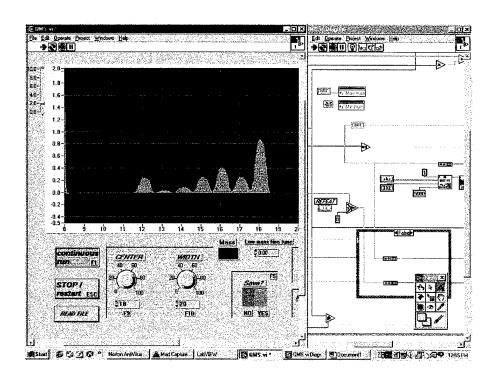
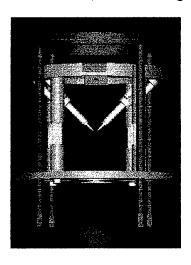


Figure 2. The developed Labview program for the QMS control allows efficient measurements of residue and process gases in the UHV chamber.

3. The Multiple Tip STM

Figure 3 shows the homebuilt STM mounting assembly with 2 of the 4 STM probes in place. This mounting assembly fits into the UHV chamber from below. Specific springs have been fabricated by using Inconel 700 wire. All four springs have exactly adjusted force constants in order to isolate the STM from the surrounding system and to avoid coupling of the resonant frequencies of the apparatus. The STM tips are mounted at 45 degrees to the axis, and at 90 degrees to each other.



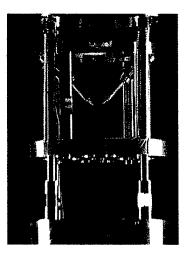
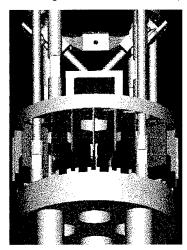


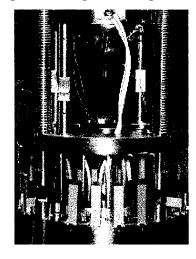
Figure 3. STM assembly and support ring. (a) Design drawing, (b) actual device.

During the development of the multiple tip STM/SEM we encountered two major problems:

- 1) Inevitable floor vibrations caused disturbing tip vibrations in the 4Hz range.
- 2) Electro-magnetic noise generated by electric building equipment disturbed high resolution SEM imaging and a smooth tip-sample approach.

The first problem was solved by incorporation an eddy current damping system in the multiple tip STM. Figure 4 shows the design and a photograph of the actual eddy current system for the multiple tip STM. It consists of 16 custom made SmCo magnets with a Curie temperature of 750 °C (Magnetic Component Engineering, INC). The circular design allows





an almost field free operation of the electron column in the center above the STM stage.

The second problem could be eliminated only partially. We are currently exploring a novel active field cancellation system.

Figure 4. UHV multiple STM with eddy current damping system. Left hand: Design drawing. Right hand side: Photograph of the actual system.

Figure 5 shows on the left-hand side one nanomanipulator in detail and on the right hand side the Windows 98 based control software. The actuators will move the tips along their axes as well as tilting the individual support base of each tip motor, thereby allowing all positions to be achieved. Computer control of all course and fine motions is currently possible, and refinements are being incorporated.

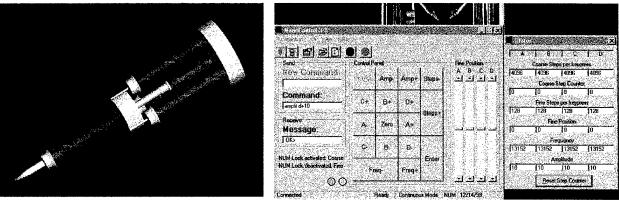
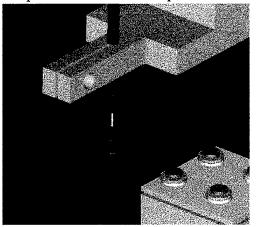


Figure 5. (a) Detailed view of the STM nano-manipulator and (b) Control software.

4. Tip Fabrication

The fabrication of sharp tips is being investigated with the use of controlled etching procedures under an optical microscope. A schematic of the electrochemical tip-etching



device is shown in Figure 6. Here a tungsten wire is lowered into a film of NaOH solution supported on the ring. A high reproducible precision could be achieved by modifying an existing toolmakers microscope in combination with a nanomotor. Control of the vertical tip position while etching has permitted the production of tips with high control and reproducibility as determined by studies involving TEM imaging.

Figure 6. Microscopic tip etching device- schematic.

An example of a sharp tip etched in our laboratory is shown in Figure 7. The tip has a diameter of less than

10 nm. This means that the closest distance of approach of two tips can be of the order of 10-20 nm when they are probing a nanometer object.

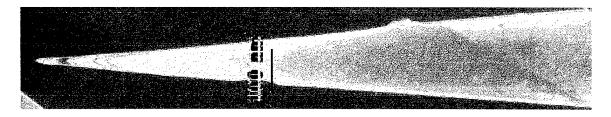


Figure 7. TEM image of tungsten tip with less than 10 nm diameter

5. SEM Measurements

5.1 Revitalization of Standard SEM

In addition to the construction and development of the nanoworkbench, we have taken a Cambridge SEM from the Department of Chemistry and revitalized the instrument to achieve about 10 nm resolution, using both the adjustment of the electron optics and also computer programs which are also used in the SEM of the nanoworkbench. The Cambridge SEM permits the rapid insertion of samples from the laboratory ambient, since it is not an ultrahigh vacuum device. Thus a wide range of nanometer size materials can be imaged in a short time, and this will be of importance in extending the nanoworkbench into many areas involving diverse materials.

Figure 8 shows an array of polystyrene spheres of 800 nm diameter which have been coated with Au by sputter deposition. The sample preparation was done in a few minutes and the image was obtained within an hour, using the Cambridge SEM.

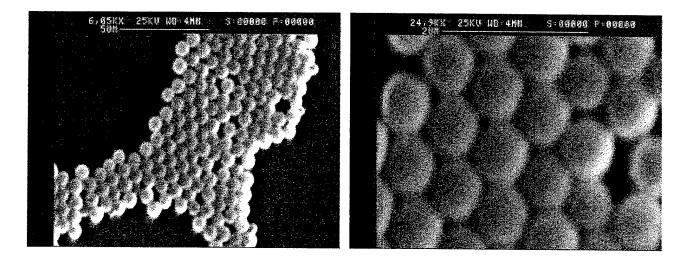
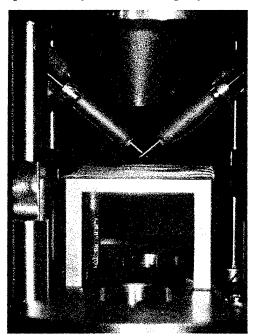


Figure 8. SEM image of 800 nm diameter polystyrene spheres, coated with Au.

5.2 UHV- SEM. Operation of the FEI Electron Column

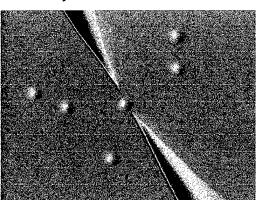
The necessary control electronics to operate the FEI electron column was provided by the FEI Company based on a rental agreement and later purchased from



other funds. Using a home-build channeltron detector, which was operated in pulse-counting mode, we tested the operation of the FEI column (Figure 9). The resolution of the column was not better than 100 nm due to floor vibrations, which could not be eliminated with the existing simple air based vibration damping. In order to solve the vibration problem we are currently constructing a rigid box holder for the complete system combined with state of the art active vibration elements.

Figure 9. The electron column mounted in the UHV chamber (only the end tip is seen) together with the two STM tips approaching each other.

Figure 10 shows two SEM images from a video documenting two STM tips approaching an isolated gold coated polystyrene (PST) particle of 500nm diameter. We succeeded for the first time to precisely position two tips under SEM control on selected PST particles in order to measure their electronic properties. This is an important step forward to explore novel nano-eletronic devices, as proposed.



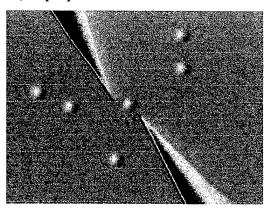


Figure 10. In-situ SEM imaging of two STM tips approaching an isolated PST particle. This images demonstrate for the first time the operation of a multiple tip STM.

6. Future Plans

The research plans follow closely those outlined in our FY 2000 DURIP proposal.

We are currently testing the multiple tip STM with additional two STM tips installed, and an improved tip exchange system. We are designing a high precision x/y/z STM/SEM stage manipulator together with a variable temperature sample-holder. This will be essential to perform the electronic transport measurement of nanoobjects. We are currently designing a multiple chamber system in which the nanoworkbench will be incorporated.

The design of the three UHV chamber system shown is shown in Figure 11. All chambers are connected with each other through a special sample transfer system. This chamber system allows ultra clean sample preparation and characterization. The multiple source MBE (molecular beam epitaxy) chamber has been designed to be equipped with several electron beam evaporators for precise deposition and doping of metals and semiconductors.

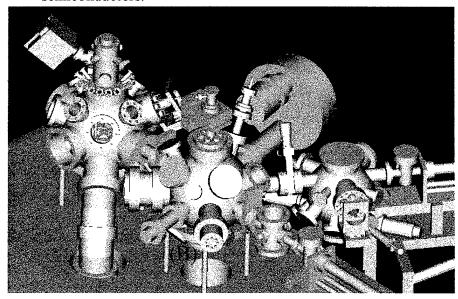


Figure 11. Three UHV chamber system. The chamber (A) on the left-hand side houses the four tip STM, the SEM and the AES detector. The chamber (B) in the center incorporates several surface analysis instruments. On the right hand side the MBE chamber (C) is shown.

7. Publications Related to This Project

- Joachim Ahner and John T. Yates Jr., "Novel Multiple-Tip STM and Nanoworkbench for Manipulating, Imaging and Analyzing of Nanostructures", Proceedings of the VW-Symposium "New Trends in Physics, Chemistry and Biology with Single Molecules", Wiesbaden, Germany, July 1999.
- J. Ahner, "Nanoworkbench for Imaging, Analysis, Manipulation and Excitation of Individual Nanostructures", MSTnews, 4, 16 (1999).